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(71) Applicant: THE PROCTER & GAMBLE CO [US/US]; One Procter & Gamble Plaza, Cincing		IY UG, ZW), Eurasian patent (A).	1, AZ, BY, KG, KZ, MD,
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#### Constitution Drive, Cincinnati, OH 45215 (US). SEIDEN, Paul; 2890 Grandin Road, Cincinnati, OH 45208 (US).

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Paul; 2890 Grandin Road, Cincinnati, OH 45208 (US).

(74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217-1087 (US).

(72) Inventors: ZIMMERMAN, Stephen, Paul; 270 Ritchie Avenue,

Wyoming, OH 45215 (US). REED, Jada, Dawn: 7501

(54) Title: EMULSIFIER-LIPID COMPOSITION

(57) Abstract

An emulatine-lipid composition comprising a monoglyceride component, a polyglycerol ester component and a fat component. The monoglyceride component is selected from mono-disperded, distilled monoglycerides or mixture theroof, and comprises and from about 2.0 % to about 30 % of the emulsifier-lipid composition. The preferred mono-displyceride of distilled monoglycerides of distilled monoglycerides of the emulsifier-lipid composition. The polyglycerol ester component comprises from about 0.5 % to about 40 % of the mixture of the component is a to about 10 givection of the polyglycerol ester component is an about 10 givection of the properties from about 10 about 10 givection units per polyglycerol ester component has a best of the polyglycerol ester component has a set of the polyglycerol ester component has a set of the polyglycerol ester component has a best of the polyglycerol ester component has been set of the polyglycerol ester component has been set of the polyglycerol ester component may be a digestible fat or non-digestible fat and comprises from about 20 % a set of the polyglycerol ester of the polyglycerol es

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# I EMULSIFIER-LIPID COMPOSITION

#### TECHNICAL FIELD

The present invention relates to emulsifier-lipid compositions. More specifically, the present invention relates to emulsifier-lipid composition particularly useful in the preparation of sheetable doughs, such as fabricated snacks, especially low-fat fabricated snacks.

#### BACKGROUND

Emulsifiers are commonly used in the preparation of doughs which are further processed by sheeting, extruding, frying, baking and other food processing activities. The composition and functionality of the emulsifier has great impact on the process feasibility of the dough and the texture of the final product. In the process of sheeting, dough compositions are formed into a sheet using various sheeting processes or equipment such as a roll mill. The dough is subjected to various stressful processes (e.g., mixing, sheeting, milling, and extrusion). During the mixing, milling and sheeting of the dough, the starch cells swell and burst releasing amylose (i.e., free starch). The amylose interacts with the water in the dough to give a cohesive, elastic dough sheet. However, if too much amylose is present in the dough composition, the dough will become sticky and adhere to the sheeting and cutting equipment. The presence of large amounts of amylose in the dough composition will also result in a rigid and stiff dough that prevents the dough from being sheeted and from expanding during frying. This results in an end food product that is dense, hard and brittle.

Emulsifiers, in particular, mono-diglycerides are generally added at low levels to the dough compositions. The emulsifier helps prevent stickiness of the dough by complexing a portion of the amylose while allowing enough stiffness and adhesiveness in the dough to form a cohesive and elastic dough sheet.

Determining which particular emulsifier to use in a particular dough composition is extremely difficult. The almost infinite number of chemical compositions available and the physical surface-active properties of the emulsifier generally preclude accurate predictions of emulsifier behavior in the sheeted dough. Further complicating the determination is the uncertainty of blending the emulsifier with other lipid systems such as other emulsifiers, triglyceride, and non-digestible fats.

Several references describe the use of emulsifiers in sheeted doughs. U.S. Patent No. 4,678,672 to Dartey et al., issued July 7, 1987, discloses a reduced calorie cracker comprising at least one emulsifier having a hydrophilic/lipophilic balance (HLB) of 11 selected from sorbitan monostearate, mono- and/or diglycerides, polyoxyethylene sorbitan fatty acid esters, and sodium stearoyl-2-lactylate. Dartey et al. also discloses that lower HLB emulsifiers such as ethoxylated mono- and diglyceride, polyglycerol esters and diacetyl tartaric acid esters may be used in combination with emulsifiers having an HLB of 11. This reference however does not disclose the specific composition of the various emulsifiers. Further the emulsifier-lipid compositions do not contain a non-digestible fat.

Although those skilled in the art use various emulsifiers in sheeted doughs as processing aids, they are not apt to encounter problems in practice when making sheetable doughs intended to be fried in non-digestible fat. For example, it has been found that when monoglycerides or mono-diglycerides having certain compositions are combined with non-digestible fat and put into a starch-based dough composition, the dough becomes less elastic. The doughs tend to change shape easily, especially during processing. During frying, the doughs may shrink. This results in a product that is dense and hard. It has also been found that when certain polyglycerol esters and non-digestible fats are combined and used in a starch-based dough formula, the dough composition is very elastic. The ability of the dough to retain its original shape after processing is increased. This results in a product that is expanded and foamy.

Conventional processing techniques also do not address the problems of combining emulsifiers and non-digestible fat nor direct the processor as to which compositions, among the infinite number of compositions, are suitable for use in fabricated snacks fried in non-digestible fat. For example, when polyglycerol esters having certain chemical compositions and/or higher levels of hydrophilic moteties are combined with non-digestible fat, phase separation can occur.

It can be seen that known methods of emulsification do not address the problem of providing an emulsifier or an emulsifier-lipid composition that will provide a sheetable, cohesive and extensible dough suitable for frying in non-digestible fat without adverse textural disadvantages.

Accordingly, it is the object of the present invention to provide emulsifier-lipid compositions.

It is another object of the present invention to provide emulsifier-lipid compositions suitable for use in fabricated snacks, especially chips fried in non-digestible fat.

It is still another object of the invention to provide an emulsifier-lipid composition comprising a combination of components having specific compositions.

The emulsifier-lipid composition of the present invention is meant to reduce the aforementioned problems related to emulsifier use, dough rheology and textural disadvantages in the finished product.

#### SUMMARY OF THE INVENTION

The present invention relates to a novel emulsifier-lipid composition comprising at least three components. This emulsifier-lipid composition comprises a specific blend of a monoglyceride component (which consists of mono-diglycerides and/or distilled monoglycerides), a polyglycerol ester component, and a fat component. The first component is a monoglyceride component which comprises from about 2.0% to about 50% of the emulsifier-lipid composition. The monoglyceride component has a high concentration (>60%) of monoglyceride. The second component is a polyglycerol ester component which

comprises from about 0.5% to about 40% of the emulsifier-lipid composition. The polyglycerol ester comprises less than 50% free polyol. The polyglycerol ester comprises from about 2 to about 10 glycerol units per polyglycerol moiety. The glycerol units have less than 40% of their hydroxyl groups esterified with myristic acid, palmitic acid, stearic acid, or mixtures of these acids. The third component is a fat component which comprises from about 20% to about 97.5% of the emulsifier-lipid composition. A non-digestible fat, if used, for all or part of the fat component preferably has properties similar to a triglyceride, for example, a polyol fatty acid polyester.

The emulsifier-lipid composition blend may be prepared by mixing together the above components in the specificd amounts. It has been found that the emulsifier blend of the present invention provides significant improvements in crispness of fabricated snacks fried in non-digestible fat. The emulsifier improves the organoleptical properties of the snack and provides a method for producing a snack having a unique structure. The improved results are not achievable with any of the individual components alone or with two-component systems using the same or similar emulsifier.

#### DETAILED DESCRIPTION

#### **Definitions**

The term "sheetable dough" as used herein is a dough capable of being placed on a smooth surface and rolled to the desired final thickness without tearing or forming holes.

The term "monoglyceride" as used herein refers to fatty acids containing one fatty acid chain attached to the glycerol. The monoglyceride component described herein is comprised of 30-98% monoglycerols, less than 2% free glycerine, and diglycerides with a small amount of free glycerine or free fatty acids.

The term "mono-diglyceride" as used herein refers to a mixture of fatty acid monoglycerides and fatty acid diglycerides, triglycerides and free glycerine and free fatty acids with a monoglyceride level greater than 30%.

The term "distilled monoglyceride" as used herein refers to a fractionated mixture of fatty acid monoglycerides with a monoglyceride level greater than 60%.

The term "polyglycerol" as used herein refers to condensed glycerol molecules, such as dimeric glycerol (diglycerol), trimeric glycerol (triglycerol), etc. Commercial polyglycerol products are normally mixtures containing glycerol in varying amounts of polymerization from monomeric glycerol up to tetrameric or higher glycerol condensates.

The term "emulsifier" as used herein refers to a single emulsifier.

All percentages are by weight unless otherwise stated.

Emulsifier-lipid composition

The emulsifier-lipid composition comprises three functional components: a monoglyceride component, a polyglycerol ester component, and a fat component.

One component of the emulsifier system is a monoglyceride component. The monoglyceride component is comprised of mono-diglycerides, distilled monoglycerides or mixtures thereof. The monoglyceride component of the emulsifier-lipid composition of the present invention may be a mixture of saturated and unsaturated glycerol esters of fatty acids typically derived from hydrogenated to non-hydrogenated vegetable oils such as soybean oil, corn oil, olive oil, sunflower oil, cottonseed oil, palm oil and like vegetable oils, and animal fats such as tallow and lard. The monoglyceride component comprises at least 30% monoglycerides. Preferably, concentrated monodiglycerides (i.e. containing >30% monoglyceride) or distilled monoglycerides are used. The more concentrated mono-diglycerides or distilled monoglycerides comprise at least about 60%, preferably from at least about 70% to at least about 98%, more preferably from at least about 80% to at least about 90% monoglyceride, with the balance being diglycerides with small amounts of triglyceride and free glycerine. Preferably the amount of free glycerine present in the mono-diglyceride component is less than about 2.0%. The amount of monoglyceride present in the mono-diglyceride or distilled monoglyceride and be determined using AOCS Cd 11-b-91 (95).

The mono-diglycerides or distilled monoglycerides useful in the present invention have an iodine value in the range of from about 2 to about 120, preferably from about 20 to about 100, more preferably from about 40 to about 80, and most preferably from about 50 to about 75. The iodine value can be determined using AOCS method Cd 1-25 (93).

Preferably the mono-diglycerides or distilled monoglyceride have a linolenic fatty acid level of less than 3.5%.

Specific mono-diglycerides or distilled monoglycerides within the scope of the present invention are commercially available. Monoglycerides suitable for use in the present invention are sold under the trade names of Dimodan® available from Danisco, New Century, Kansas and DMG 70, available from Archer Daniels Midland Company, Decaus, Illinois.

The monoglyceride component comprises from about 2.0% to about 50%, preferably from about 5.0% to about 40%, more preferably from about 10% to about 30%, and most preferably from about 12% to about 25% of the total emulsifier-lipid composition.

The second component of the emulsifier-lipid composition is a polyglycerol ester. Examples of polyglycerol ester include decaglycerol decaoleate, triglycerol monostearate, octaglycerol monostearate, and octaglycerol mono-palmitate. These materials are normally not obtained in pure form, but are generally the reaction products of an esterification between a preselected cut of polyglycerols and desired saturated fatty acids. The result is a distribution of polyglycerol mono-ester and higher-esters determined by ratio of reactants and reaction conditions.

The polyglycerol esters of the present invention are specifically tailored by controlling the hydrophilic-tipophilic balance (HLB) of the polyglycerol esters. This is done by controlling the balance of esterified to unesterified hydroxyl groups during the process of esterfication. With an increasing number of hydroxyl groups esterified, the polyglycerol ester becomes progressively more lipophilic. This hydrophilic-lipophilic balance of the polyglycerol ester is important in preparing polyglycerol ester for use in sheeted douehs.

Unesterified polyglycerols, long chain polyglycerol monoesters, and diesters and tri-esters of diglycerols and triglycerols should be limited in the polyglycerol ester component of the present invention. Unreacted polyglycerol (i.e. unesterfied) retained in the finished esters have little or no emulsifier functionality, but because of their more polar nature are less soluble in non-digestible lipids leading to phase separation and a non-homogenous emulsifier-lipid composition.

The smaller chained polyglycerol monoesters are very functional components of the polyglycerol esters in the polyglycerol ester component of the emulsifier-lipid composition and thus their concentration should be relatively high compared to other ester moieties. The di- and triesters of di- and triglycerols are too lipophilic and may also have a deleterious effect on the finished snack product. Saturated diglycerides (e.g. dipalmitin, distearin) and the cylic diglycerol esters are deleterious emulsifier components and therefore their concentrations should be minimized in the polyglycerol esters. Preferably the polyglycerol esters of the present invention comprise less than 5% cylic diglycerol esters and less than 5% diglycerides.

Polyglycerol esters can be purified through fractionation, molecular distillation or solvent crystallization. The fractionated polyglycerol esters are more functional and can be used at lower concentration.

The composition of the polyglycerol ester can be determined by Supercritical Fluid Chromatography described in the Analytical Methods section herein below.

The polyglycerol esters suitable for use in the present invention comprise less than 50%, preferably from about 2.0% to about 40%, and more preferably from about 5.0% to about 25% free glycerine; from about 5.0% to about 60%, preferably from about 15% to about 50%, more preferably from about 15% to about 40% monoester. The polyglycerol ester of the present invention additionally has from about 2 to about 10 glycerol units per polyglycerol moiety wherein the glycerol units have less than 40%, preferably from about 18% to about 33%, more preferably from about 20% to about 30% of their hydroxyl groups esterified with myristic acid, palmitic acid, stearic acid or mixturest thereof.

The polyglycerol ester component comprises from about 0.5% to about 40%, preferably from about 1.0% to about 35%, more preferably from about 1.5% to about 30% and most preferably 2.0% to about 25% of the total emulsifier-lipid composition.

Polyglycerol esters suitable for use in the present invention are sold under the trade name Lonza Polyaldo®.

The third component of the emulsifier-lipid composition of the present invention is a fat. The terms "fat" and "oil" are used interchangeably herein unless otherwise specified. The terms "fat" or "oil" refer to edible fatty substances in a general sense, including natural or synthetic fats and oils consisting essentially of triglycerides, such as, for example soybean oil, corn oil, cottonseed oil, sunflower oil, palm oil, coconut oil, canola oil, fish oil, lard and tallow, which may have been partially or completely hydrogenated as well as non-toxic fatty materials having properties similar to triglycerides, herein referred to as non-digestible fats, which materials may be partially or fully indigestible. Reduced calorie fats and edible non-digestible fats, oils or fat substitutes are also included in the term.

The term "non-digestible fat" refers to those edible fatty materials that are partially or totally indigestible, e.g., polyol fatty acid polyesters, such as OLEAN®.

Particularly preferred are non-digestible fats such as those described in U. S. Patent Nos. 3,600,186 to Mattson et al., issued May 12, 1970; 4,005,195 to Jandacek, issued January 25, 1977; 4,005,196 to Jandacek et al., issued January 25, 1977; 4,034,083 to Mattson, issued July 5, 1977; and 4,241,054 to Volpenhein et al., issued December 23, 1980, all of which are incorporated by reference.

By "polyol" is meant a polyhydric alcohol containing at least 4, preferably from 4 to 11 hydroxyl groups. Polyols include sugars (i.e., monosaccharides, disaccharides, and trisaccharides), sugar alcohols. other sugar derivatives (i.e., alkyl glucosides), polyglycerols such as diglycerol and triglycerol. pentaerythritol, sugar ethers such as sorbitan and polyvinyl alcohols. Specific examples of suitable sugars are mannose, galactose, arabinose, xylose, ribose, apiose, rhamnose, psicose, fructose, sorbose, tagatose, ribulose, xylulose, and enthrulose. Oligosaccharides suitable for use herein include, for example, maltose, kojibiose, nigerose, cellobiose, lactose, melibiose, gentiobiose, turanose, rutinose, trehalose, sucrose and raffinose. Polysaccharides suitable for use herein include, for example, amylose, glycogen, cellulose, chitin, inulin, agarose, zylans, mannan and galactans. Although sugar alcohols are not carbohydrates in a strict sense, the naturally occurring sugar alcohols are so closely related to the carbohydrates that they are also preferred for use herein. Natural sugar alcohols which are suitable for use herein are sorbitol, mannitol, and galactitol. Particularly preferred classes of materials suitable for use herein include the monosaccharides, the disaccharides and sugar alcohols. Preferred unesterified polyols include glucose, fructose, glycerol, alkoxylated polyglycerols, sugar ethers, and linked alkoxylated glycerines as described in U.S. Patent No. 5,516,544 to Sekula et al., issued June 14, 1996, incorporated by reference. A particularly preferred polyol is sucrose. Preferred alkoxylated glycerols are described in the following U.S. Patents. incorporated by reference herein; U.S. 5,273,772 to Cooper, issued Dec. 28, 1993; U.S. 5,288,884 to Cooper, issued Feb. 22, 1994; U.S. 5,298,637 to Cooper, issued March 29, 1994; U.S. 5,362,894 to Handwerker et al., issued Nov. 8, 1994; U.S. 5,374,446 to Ferenz et al., issued Dec. 20, 1994; U.S. 5,387,429 to Cooper, issued Feb. 7, 1995; U.S. 5,427,815 to Ferenz, issued June 27, 1995; U.S. 5,466,843 to Cooper, issued Nov. 14, 1995; U.S. 5,516,544; U.S. 5,589,217 to Mazurek, issued Dec. 31, 1996; and U.S. 5,597,605 to Mazurek, issued Jan. 28, 1997. More preferred alkoxylated glycerines are

linked alkoxylated glycerines and are described in the following patents, previously incorporated herein, 5,374,446; 5,427,815; and 5,516,544. Especially preferred alkoxylated glycerines are those described in U.S. Patent Number 5,516,544, previously incorporated by reference.

By "polyol fatty acid polyester" is meant a polyol having at least 4 fatty acid ester groups. Polyol fatty acid esters that contain 3 or less fatty acid ester groups are generally digested in, and the products of digestion are absorbed from, the intestinal tract much in the manner of ordinary triglyceride fats or oils, whereas those polyol fatty acid esters containing 4 or more fatty acid ester groups are substantially non-digestible and consequently non-absorbable by the human body. It is not necessary that all of the hydroxyl groups of the polyol be esterified, but it is preferable that disaccharide molecules contain no more than 3 unesterified hydroxyl groups for the purpose of being non-digestible. Typically, substantially all, e.g., at least about 85%, of the hydroxyl groups of the polyol are esterified. In the case of sucrose polyesters, typically from about 7 to 8 of the hydroxyl groups of the polyol are esterified.

The polyol fatty acid esters typically contain fatty acid radicals typically having at least 4 carbon atoms and up to 26 carbon atoms. These fatty acid radicals can be derived from naturally occurring or synthetic fatty acids. The fatty acid radicals can be saturated or unsaturated, including positional or geometric isomers, (e.g., cis- or trans- isomers) and can be the same for all ester groups, or can be mixtures of different fatty acids.

Liquid non-digestible oils can also be used in the practice of the present invention. Liquid nondigestible oils which have a complete melting point below about 37°C include liquid polyol fatty acid polyesters (see Jandacek; U.S. Patent 4,005,195; issued January 25, 1977); liquid esters of tricarballylic acids (see Hamm; U.S. Patent 4,508,746; issued April 2, 1985); liquid diesters of dicarboxylic acids such as derivatives of malonic and succinic acid (see Fulcher; U.S. Patent 4,582,927; issued April 15, 1986); liquid triglycerides of alpha-branched chain carboxylic acids (see Whyte; U.S. Patent 3,579,548; issued May 18, 1971); liquid ethers and ether esters containing the neopentyl moiety (see Minich; U.S. Patent 2,962,419; issued Nov. 29, 1960); liquid fatty polyethers of polyglycerol (See Hunter et al; U.S. Patent 3,932,532; issued Jan. 13, 1976); liquid alkyl glycoside fatty acid polyesters (see Meyer et al; U.S. Patent 4,840,815; issued June 20, 1989); liquid polyesters of two ether linked hydroxypolycarboxylic acids (e.g., citric or isocitric acid) (see Huhn et al; U.S. Patent 4,888,195; issued December 19, 1988); various liquid esterfied alkoxylated polyols including liquid esters of epoxide-extended polyols such as liquid esterified propoxylated glycerins (see White et al; U.S. Patent 4,861,613; issued August 29, 1989; Cooper et al; U.S. Patent 5,399,729; issued March 21, 1995; Mazurek; U.S. Patent 5,589,217; issued December 31, 1996; and Mazurek; U.S. Patent 5,597,605; issued January 28, 1997); liquid esterified ethoxylated sugar and sugar alcohol esters (see Ennis et al; U.S. Patent 5,077,073); liquid esterified ethoxylated alkyl glycosides (see Ennis et al; U.S. Patent 5,059,443, issued October 22, 1991); liquid esterified alkoxylated polysaccharides (see Cooper; U.S. Patent 5,273,772; issued December 28, 1993); liquid linked esterified alkoxylated polyols (see Ferenz; U.S. Patent 5,427,815; issued June 27, 1995 and Ferenz et al; U.S. Patent

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5,374,446; issued December 20, 1994); liquid esterfied polyoxyalkylene block copolymers (see Cooper; U.S. Patent 5,308,634; issued May 3, 1994); liquid esterified polyethers containing ring-opened oxolane units (see Cooper; U.S. Patent 5,389,392; issued February 14, 1995); liquid alkoxylated polyglycerol polyesters (see Harris; U.S. Patent 5,389,371; issued March 21, 1995); liquid partially esterfiede polysaccharides (see White; U.S. Patent 4,959,466; issued September 25, 1990); as well as liquid polydimethyl siloxanes (e.g., Fluid Silicones available from Dow Corning). All of the foregoing patents relating to the liquid nondigestible oil component are incorporated herein by reference. Solid non-digestible fats or other solid materials can be added to the liquid non-digestible oils to prevent passive oil loss. Particularly preferred non-digestible fat compositions include those described in U.S. 5,490,995 issued to Corrigan, 1996, U.S. 5,480,667 issued to Corrigan et al, 1995, U.S. 5,451,416 issued to Johnston et al, 1995 and U.S. 5,422,131 issued to Elsen et al, 1995. U.S. 5,419,925 issued to Selden et al, 1995 describes mixtures of reduced calorie triglycerides and polyol polyesters that can be used herein but provides more digestible fat than is typically preferred.

The preferred non-digestible fats are fatty materials having properties similar to triglycerides such as sucrose polyesters. OLEAN®, a preferred non-digestible fat, is made by The Procter and Gamble Company. These preferred non-digestible fats are described in Young; et al., U.S. Patent 5,085,884, issued February 4, 1992, and U. S. Pat. 5,422,131, issued June 6, 1995 to Elsen et al.

The fat component of the emulsifier-lipid composition of the present invention comprises from about 20% to about 97.5%, preferably from about 65% to about 90%, and more preferably from about 70% to about 85% of the emulsifier-lipid composition.

The emusifier-lipid composition of the present invention has unique thermal properties, onset of crystallization and endothermic area that relate to the performance of the emulsifier in dough systems. Determination of the thermal properties using a DSC is well known in the art. Briefly, the onset of crystallization and endothermic area is determined by using a Differential Scanning Calorimeter (DSC), Perkin Elmer Model # 7. The emulsifier-lipid composition is placed into a DSC pan and crimped. The sample is scanned at a rate of about 5°C/min. The temperature is raised beyond the melting point of the emulsifier-lipid blend (indicated by an exothermic peak and return to baseline). After the melting, the sample is then cooled at a rate of about -5°C/min, until a temperature of 0°C is reached. The point at which the endotherm begins to move away from the baseline is the onset of crystallization. The endothermic area is also used to determine how the emulsifier will perform in doughs. The endothermic area between the dough making and onset of crystallization temperature is used for this purpose.

The emulsifiers of the present invention preferably have an onset of crystallization in the range of from about 100°F (37.7°C) to about 135°F (57.2°C). The onset of crystallization temperature and the amount of crystallization that can occur relates to the ability of the emulsifier or emulsifier blend to disperse homogeneously throughout the dough and adequately disperse the water present in dough compositions. The endothermic area (i.e., the area shown above typical dough making sheet formation temperature) is the measurement used to predict how the emulsifier will behave during sheeting. The snacks of the present invention have an endothermic area above 108°F (42.4°C) of less than about 150 millijoules, preferably less than about 125 millijoules, more preferably from about 1 to about 80 millijoules, more preferably from about 2 to about 40 millijoules, and most preferably from about 4 to about 10 millijoules.

The droplet size of the emulsifier-lipid composition has been found to be an indication of the type of internal product structure and void size and area that will be present in the finished product. Since this void size is also related to the texture of the resulting fried snack, the type and size of oil droplets formed are important. The method for determining the oil droplet size is described herein. The oil droplets of the emulsifier-lipid composition of the present invention are small and uniform in size. This enables the emulsifier-lipid composition to disperse homogeneously throughout the dough composition. When dispersed in water, the emulsifier-lipid composition of the present invention tends to aggregate in clusters of several hundred droplets. At 100x magnification, the aggregate droplet size is preferably less than 3.0 cm<sup>2</sup>, preferably less than 1.5cm<sup>2</sup>, and more preferably between 0.01 to 0.05 cm<sup>2</sup>

The emulsifier-lipid composition of the present invention is generally used in farinaceous fabricated snack such as potato chips, corn chips, tortilla chips, half products, and extruded snacks at a level of from about 0.5% to about 8.0% based on the weight of the dough.

Preparation

The emulsifier-lipid composition of the present invention is prepared by melt-blending and mixing the components until a homogeneous liquid is obtained. Melt-blending may be accomplished by individually maintaining or raising the temperatures of the components to a point above their respective melting temperatures and then thoroughly blending, or by mixing the components at room temperature and raising the temperature of the mixture to at least the melting point of the highest-to-melt component followed by thorough blending to form a homogeneous liquid.

## ANALYTICAL METHODS SUPERCRITICAL FLUID CHROMATOGRAPHY

A sample of polyglycerol ester is first silylated to derivatize any unreacted hydroxyl groups. The silylated sample is then injected into the supercritical fluid chromatograph (SFC). The esters are separated by degree of esterification on a DBI capillary column and detected by a flame ionization detector. The distribution of esters is calculated by peak area.

#### Equipment and Conditions

SFC: Lee scientific series 6000 supercritical fluid chromatograph or equivalent; SFC Conditions:

- Capillary Column

  DB1, 0.2 u film, 50 u ID, 10 m. J&W Scientific
- B) <u>Temperatures</u> Oven - 90°C

Detector - 400°C

- C) <u>Pressure Program</u> Pressure, Time 125: 375, 25: 375, 10: 0, 0
- D) <u>CO2</u> SFC grade, Scott Specialty Gases
- E) <u>Hydrogen</u>
  Approximately 30 mL / minute
- F) <u>Air</u>
  Approximately 300-350 mL / minute
- G) <u>Auxiliary Gas (Nitrogen)</u>
   Approximately 25 mL / minute
- H) Syringe for SFC injection
  50 ul Hamilton
- Vials
   2 or 4 dram Kimble Glass Fischer Scientific #03-340-1C
- J) <u>Hot Plate</u>
- 90°C K) Filter
  - 0.45 u Alltech Associates #2092
- L) <u>Disposable Syringe</u>

  3.0 mL Fisher Scientific #14-823-39

BSTFA (bis(TrimethylsilyI)-trifluoroacetamide) Supelco, Inc. #3-3027; TMSI (TrimethylsilyIimidazole) Supelco, Inc. #3-3068; Pyridine ACS Grade MCB #PX2020-01

#### Analyzing Standard

The sample is melted completely and mixed well. A disposable pipet is used to weigh 80-100 mg of sample into a four dram vial. The sample weight is recorded. 1 mL of Pyritine and 1 mL of TMSI/BSTFA solution (mixed 5:1) is added to the vial. The vial is capped and heated on the hot plate at 90°C for 15 minutes. The sample is allowed to cool. A 0.45-micron filter is placed on the end of a 3-cc

disposable syringe. The derivatized standard is poured into the disposable syringe and filtered into a GC vial. The sample is injected into the Supercritical Fluid Chromatograph.

#### Emulsion Droplet Size Method

- 1. Fifty (50) grams of emulsifier composition are heated to a complete melt at 155°F.
- An equal amount of water, 50 grams, is added to the emulsifier pre-heated to 170°F.
- 3. The water and emulsifier blend are mixed using a stainless steel whisk, mixing for 30 seconds, adding water in 50 gram increments to the solution until the water is 5.5 times the amount of emulsifier (275 grams); or all of the water and emulsifier are mixed using a lab scale shear mixer (ex. Janke and Kunkel SD-45 at a setting of 7) for 60 seconds until the two components are thoroughly combined.
- 4. A sample of the emulsion is placed on a new, clean microscope slide with a cover slip and evaluated at a magnification of 100x using a Zeiss light microscope with polarized light filters and photographic attachments
- 5. A picture of the emulsion is taken within 120 seconds of formation.
- Droplet size is measured using a 2.6 cm X 2.6 cm grid with 0.2 cm line increments for Polaroid pictures sized 11.5 cm X 8.9 cm. Any magnification of pictures will require comparable magnification of grid size.

The following examples are submitted for a better understanding of the invention and are not intended to limit the scope of the present invention. In the examples, emulsifier-lipid compositions are added at the level indicated to fabricated snack compositions.

#### EXAMPLE 1

The following composition is used to make an emulsifier-lipid composition:

Ingredient	Wt. %
Non-digestible fat	85.00
Polyglycerol ester (PGE)	2.25
Distilled Monoglyceride (DMG)	12.75

The PGE is a hexaglycerol monoester of palmitic and stearic acids available from Lonza, Fair Lawn, NJ as Polyaldo with an HLB of about 10, a monoester level of about 28%, a saponification value of 100±5, a hydroxyl value of 415±30 and a free polyol level less than 22%. The DMG has a monoester level of  $\geq$  90% and an iodine value between 60-70 and is sold under the tradename of Dimodan-OK, available from Danisco, New Century KS. The non-digestible fat is OLEAN®, available from The Procter & Gamble Company, Cincinnati, OH.

Chemical Composition of Polyglycerol Esters

Components	Percentage
Monoglycerol monopalmitate	1.7
Diglycerol monopalmitate	7.3
Triglycerol monopalmitate	8.8
Tetraglycerol monopalmitate	4.6
Pentaglycerol monopalmitate, Monoglycerol dipalmitate	5.6
Total Monopalmitate	28.0
Diesters	25.9
Triesters	16.6
Tetraesters	6.1
Pentaesters	2.4
Free Polyol	21.1

The physical properties of the emulsifier system are:

Property	Value
Onset of Crystallization, C	48 (118°F)
Area of Crystallization, mj	0.84
Complete Melt Point, C	67 (153 F)
Oil Droplet Agglomerate Size Range, cm <sup>2</sup> @ 100x magnification	0.01-0.33

EXAMPLE 2

The following composition is used to make an emulsifier-lipid composition:

Ingredient	Wt %
Non-digestible fat	85.00
Polyglycerol ester (PGE)	2.25
Distilled Monoglycerid(DMG)	12.75

The PGE is predominantly diglycerol monopalmitate available from Lonza, Fair Lawn, NJ with an HLB of less than 10, a monoester level of about 43%, a saponification value of 124, a hydroxyl value of 402, and a free polyol level of about 17%. The DMG has a monoester level of ≥ 90% and an iodine value between 60-70 and is sold under the tradename of Dimodan-OK, available from Danisco, New Century, KS. The non-digestible fat is OLEAN®, available from The Protete & Gamble Company, Cincinnati, OH.

Chemical Compositions of Polyglycerol Esters

Components	Percentage
Monoglycerol monopalmitate	0.4
Diglycerol monopalmitate	25.7
Triglycerol monopalmitate	8.8
Tetraglycerol monopalmitate	3.9
Pentaglycerol monopalmitate, Monoglycerol dipalmitate	4.3
Total Monopalmitate	43.1
Diesters	30.5
Triesters	8.0
Tetraesters	1.8
Pentaesters	0.0
Free Polyol	16.6

#### EXAMPLE 3

The following composition was used to make an emulsifier system:

Ingredient	Wt %
Non-digestible fat	85.00
Polyglycerol ester (PGE)	2.25
Distilled Monoglyceride (DMG)	12.75

The PGE is predominantly diglycerol monopalmitate available from Lonza, Fair Lawn, NJ with an HLB of less than 10, a monoester level of about 44%, a saponification value of 133, a hydroxyi value of 378, and a free polyol level of less than 13%. The DMG available from Danisco as Dimodan-OK has a monoester level of ≥ 90% and an iodine value between 60-70. The OLEAN is available from The Procter & Gamble Company, Cincinnati, OH.

Chemical Composition of Polyglycerol Esters

Composition of Corpgition Listers		
Percentage		
0.3		
24.8		
11.3		
4.9		
2.8		
44.1		
31.8		
9.6		
1.6		
0.4		
12.5		

#### What is claimed is:

- An emulsifier-lipid composition characterized in that it comprises:
  - a) from about 2.0% to about 40% of a monglyceride component comprising;
    - (i) from about 30% to about 98% monoglycerides;
    - (ii) less than 2% free glycerine:
    - (iii) the balance being diglycerides with small amounts of triglycerides:
  - from about 0.5% to about 40% of a polyglycerol ester component comprising;
    - (i) less than 50% free polyol glycerine;
    - (ii) from about 2 to about 10 glycerol units per polyglycerol moiety wherein less than 40% of their hydroxyl groups are esterified with myristic acid, palmitic acid, stearic acid, or mixtures thereof; and
  - from about 60% to about 97.5% fat.
- The emulsifier-lipid composition according to Claim 1 wherein the polyglycerol ester comprises less than 30% glycerine.
- The emulsifier-lipid composition according to any one of the preceding claims wherein the monoglyceride component comprises from 10% to 40%, preferably 12% to 30% of the emulsifier-lipid composition.
- 4. The emulsifier-lipid composition of according to any one of the preceding claims wherein the polyglycerol ester component comprises from 1.0% to 35%, preferably 1.5% to 30% of the emulsifier-lipid composition.
- The emulsifier-lipid composition according to any one of the preceding claims wherein the polyglycerol ester component has less than 5.0% cyclic polyglycerol esters and less than 5.0% diglycerides.
- The emulsifier-lipid composition according to any one of the preceding claims wherein the monoglyceride component comprises from 70% to 90% monoglycerides.
- The emulsifier-lipid composition according to any one of the preceding claims wherein the polyglycerol ester component has from 10% to 33% hydroxyl groups esterified.

 The emulsifier-lipid composition according to any one of the preceding claims wherein the fat is a non-digestible fat.

#### INTERNATIONAL SEARCH REPORT

Intern at Application No PCT/US 98/21969

A. CLASSIFICATION OF SUBJECT MATTER IPC 6 A21D2/16 A23D9/013 A23L1/308

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC 6 A21D A23D A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
x	US 4 732 767 A (SEIDEN PAUL ET AL) 22 March 1988 see column 2, line 61 - column 5, line 22 see example 1 see claim 1	1-7
х	"LOW CALORIE PUFF PASTRY MARGARINE" RESEARCH DISCLOSURE, vol. 343, 1 November 1992, page 823 XP000324084 see the whole document	1
Y	EP 0 059 507 A (PROCTER & GAMBLE) 8 September 1982 see page 7, line 6 - page 8, line 3 see page 8, line 21 - page 10, line 18 see claims 1-8,10	1-8

X Further documents are listed in the continuation of box C.	X Patent family members are listed in annex.
* Special categories of cited documents :	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in contlict with the application but cited to understand the principle or theory underlying the invention.
"E" earlier document but published on or after the International	"X" document of particular relevance; the claimed invention
"L" document which may throw doubte on priority claim(s) or which is cited to establish the publication date of another	cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
citation of other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the
"O" document referring to an oral disclosure, use, exhibition or other means	document is combined with one or more other such docu-
"P" document published prior to the international filing date but later than the priority date claimed	ments, such combination being obvious to a person skilled in the art.  "&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report
23 February 1999	10/03/1999
Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentiaan 2 NL - 2230 HV Rijswijk Tel. (+31-70) 340-2040, Tk. 31 651 epo nl, Fax: (+31-70) 340-3016	Dekeirel, M

## INTERNATIONAL SEARCH REPORT

Intern al Application No PCT/US 98/21969

C.(Continu	ation) DOCUMENTS CONSIDERED TO BE RELEVANT	FC1/US 98/21969
Category *	Citation of document, with indication, where appropriate, of the relevant passages	
	passages	Relevant to claim No.
Y	WO 92 17077 A (CURTICE BURNS INC) 15 October 1992 see examples I,II,,IV see claims 1,6,10,11,13	1-8
A	PATENT ABSTRACTS OF JAPAN vol. 017, no. 468 (C-1102), 26 August 1993 -å JP 05 115242 A (ASAHI DENKA KOGYO KK), 14 May 1993 see abstract	1
A	US 3 914 453 A (GAWRILOW ILIJA) 21 October 1975 see column 2, line 11 - line 29 see column 4, line 60 - line 65 see column 6, line 23 - line 48 see column 10, line 63 - column 11, line 30 see claims 1,5-7,10	1
A	US 5 045 337 A (EL-NOKALY MAGDA ET AL) 3 September 1991 see column 4. line 7 - column 6, line 20 see column 7, line 26 - line 52	1,8
P,A	WO 98 00037 A (PROCTER & GAMBLE) 8 January 1998 see page 8, paragraph 3 - page 9, last paragraph	1,8

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